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Investigation of Lightning and EMI Shielding Properties of SWNT Buckypaper Nanocomposites

(Final Report for Grant # FA9550-04-1-0349)

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Abstract

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In this exploratory research project, a unique technical approach was developed to incorporate single-walled carbon nanotubes (SWNTs) buckypaper materials into conventional fiber-reinforced and foam composite structures for improved EMI and lightning strike properties. The research team, comprising of researchers from Florida Advanced Center for Composite Technologies (FAC²T) and Lockheed Martin Missiles and Fire Control, characterized both of the fiber-reinforced composites and foam structures with a surface layer of SWNT nanocomposite. These nanocomposites were manufactured using the buckypaper/resin infiltration technique developed at FAC²T to create conducting surfaces on the composite structures for improved EMI and lightning strike protection properties of composite structures. Buckypaper materials are thin preformed sheets of well-controlled and dispersed porous SWNT networks produced using a multiple-step process of dispersion and filtration of nanotube suspensions. This process can produce nanocomposites with uniform, controllable nanotube alignment and high nanotube loading. These features are critical for constructing directional conducting paths in the composites for EMI and lightning strike resistance applications. These resininfiltrated buckypaper materials can be easily incorporated into conventional manufacturing processes of fiber-reinforced composites, such as compression molding, vacuum bagging, RTM and VARTM, to effectively change the surface conductivity of composites without visible weight increase.

Four types of the EMI/lightning strike test composite samples with SWNT buckypapers were produced. Each sample had two layers of random or magnetically aligned buckypapers 15~25 µm thick. Each sample size was 6" x 4" x 1/8" with approximately 700mg purified SWNTs covering its surface. Lockheed Martin Missiles and Fire Control – Orlando conducted the EMI and lightning striking tests. The results shows that the foam structures with the random nanotube buckypaper surface can achieve as much as 26 dB of EMI attenuation over the test range of 455 to 500 MHz, compared to the control panel of pure foam structure. This is remarkable considering the amount of nanotubes was less than 700 mg per 6" x 4" test panel. The results also show that the random buckypaper samples have better EMI shielding properties. However, a slight reduction of EMI shielding occurred in the carbon fiber composites with the buckypaper surface, compared to the control panel. This reduction is inconclusive due to a very small number of test samples. For the lightning strike resistance, no visible improvement was observed with the use of buckypapers.

This project is the first attempt to explore and understand the EMI and lightning strike resistance properties of nanocomposites with controlled nanostructures, desired nanotube orientation and high SWNT loading. Further investigations to improve electrical conductivity of buckypaper composite surface layer are recommended to demonstrate the exceptional electrical properties of SWNTs in composite structures.

1. Introduction

Recent investigations have shown that SWNTs possess exceptionally high elastic properties, as well as large elastic and fracture strain sustaining capabilities, exceeding those of any existing reinforcement materials used in composites. More importantly, SWNTs can be either pure metallic or semi-conducting depending on their construction chirality. They exhibit thermal conductivity about twice as high as diamond, and an electric-current-carrying capacity 1000 times higher than copper wires. SWNTs also have nanoscale dimension, similar to resin molecules. Thus, large quantities of SWNTs are much easier to form effective conducting paths in composites to protect against electromagnetic interference (EMI) and lightning strikes. SWNTs also possess exceptional mechanical properties and very lightweight. Thus, SWNT nanocomposites are very promising candidates for developing the next generation of high performance materials for protecting against EMI and lightning strikes.

This project is the first attempt to explore and understand the EMI and lightning strike resistance properties by incorporating SWNTs into current composite structures by using buckypaper/resin infiltration technique. This approach could effectively produce a thin and lightweight SWNT buckypaper nanocomposites layer on the surface of conventional composite structures. The buckypaper composites have controlled nanostructures, desired nanotube orientation and high SWNT loading, which are critical for improved EMI and lightning strike resistance performance.

2. Experimental Design

For the research, the research team manufactured both SWNT buckypapers and composite samples. Both random and magnetically aligned buckypapers 9" x 8" and 15~25 µm thick were produced using the filtration technique and custom-made filters [1~5] developed at FAC²T. The magnetically aligned buckypapers were fabricated using a 5 Tesla superconducting magnet, which resulted in significant operating cost savings compared to using a conventional 5 Tesla DC resistive magnet. Four types of the designed EMI/lightning strike testing composite samples were successfully produced: 1) random buckypaper/ROHACELL PMI foam panels; 2) magnetically-aligned buckypaper/ROHACELL PMI foam panels; 3) random buckypaper/carbon fiber composite panels; and 4) magnetically aligned buckypaper/carbon fiber composite panels. Two layers of resin-impregnated buckypapers were cured on the surface of the foam or carbon fiber composite substrates using a vacuum bagging process. The resin system used in the research was EPON862/ CURE EPI W. Each sample size was 6" x 4" x \frac{1}{8}" consisting of approximately 700mg purified SWNTs covering its surface. Lockheed Martin Missiles and Fire Control – Orlando conducted the EMI and lightning strike tests.

3. Results and Discussion

3.1 Sample Fabrication

The research team used custom-made filters to fabricate large, good quality buckypaper materials which are in turn used in preparing the samples. The nanotube materials used in the research were BuckyPearls TM , purified SWNTs from Carbon Nanotechnologies Inc. (CNI). Well-dispersed and stable SWNT aqueous suspensions were prepared by sonicating and adding a selected surfactant using a multiple-step dispersion procedure, previously developed by the research team [1~5]. The prepared suspension had a nanotube concentration of 40 mg/liter. The suspension was filtrated through a 0.45 μ m pore filter to produce random buckypapers. The filter setup and the produced buckypaper are shown in Figure 1. The nanostructure of the produced random buckypaper is shown in Figure 2, showing the clear nanotube network of a random buckypaper.

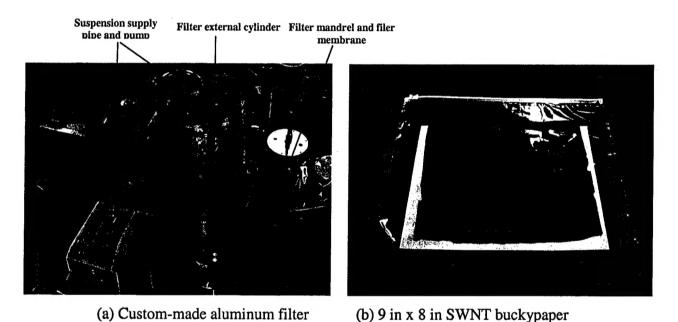
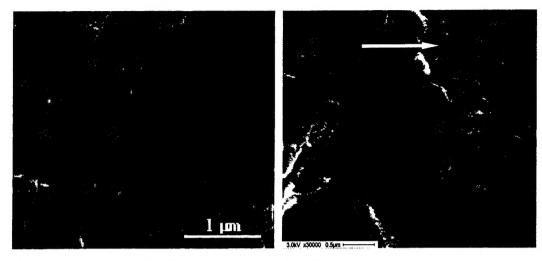


Figure 1. Custom-made filter and produced SWNT buckypaper



(a) Tube network of random buckypaper

(a) Design structure

(b) Tube network of aligned buckypaper (arrow indicating the alignment direction)

Figure 2. Nanostructures of random and magnetically aligned SWNT buckypaper

The magnetically aligned buckypapers were produced in a 5 Tesla superconducting magnet, shown in Figure 3. Compared to conventional DC resistive magnets, the superconducting magnet can achieve significant operating cost savings for producing magnetically aligned buckypaper materials. A more detailed operating cost analysis is provided in Figure 4. The cost analysis shows that the monthly cost for using a superconducting (SC) magnet is approximately 5% of that of a conventional DC resistive magnet. Furthermore, the SC magnet was available at any time, while the DC magnet is only available 7 hours a day. This cost analysis indicates that using a SC magnet could be an affordable manufacturing technique available to the government or industry for fabricating magnetically aligned SWNT buckypaper materials.

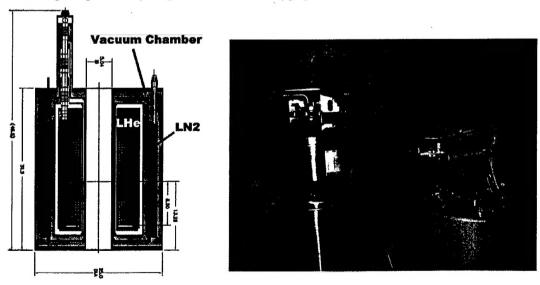


Figure 3. Structure and cooling down of the 5 Tesla superconducting magnet

(b) Cooling down using liquid helium

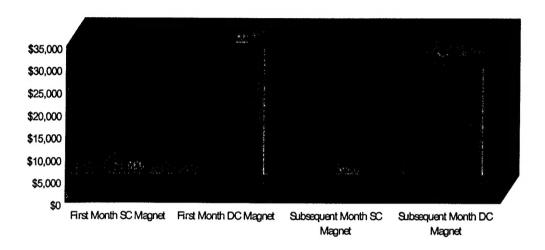
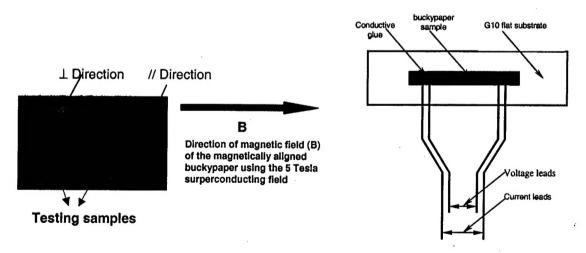


Figure 4. Operating cost analysis of both 5 Tesla superconducting and DC resistive magnet

In the research, five magnetically aligned buckypapers were successfully produced using the SC magnet. The nanostructure of the aligned buckypaper can be seen in Figure 2(b). A very fine network of the aligned nanotubes can be seen in the SEM image. The preferred tube orientation of the aligned buckypaper was also revealed in the image. To clearly quantify the tube alignment in the buckypapers, the anisotropy ratios were calculated based on the measurements of electrical resistivity perpendicular and parallel to tube alignment direction (B) in the aligned buckypaper, as shown in Figure 5.

Electrical Resistivity Anisotropy = $\frac{\text{Resistivity perpendicular to B}}{\text{Resistivity parallel to B}}$



(a) Sampling from the buckypaper (b) Setup of four-probe resistivity measurement

Figure 5. Electrical anisotropy measurements of the aligned buckypapers

The anisotropy ratio of electrical resistivity is a good indication of the nanotube alignment in the buckypapers. A four-probe method was adopted to measure the electrical resistivity perpendicular and parallel to the tube alignment direction of the magnetically aligned buckypapers. The results in Table 1 show the anisotropy ratios of electrical resistivity were around 3.06 to 3.49, which further indicates nanotube alignment existing in the magnetically aligned buckypapers. SWNT alignment in composites is critical for constructing directional paths of electrical current for both EMI and lightning protection features in composites.

In the research, four types of testing composite samples were fabricated. The sample list is shown in Table 2. Two layers of the produced buckypapers were cured on the surface of the foam or carbon fiber composite substrates using a vacuum bagging process. Each sample was 6" x 4" x \(^1/8\)" with approximately 700mg purified SWNTs covering its surface. In the buckypaper composite layer, the SWNT content was about 50w%. The foam materials used in the research were ROHACELL PMI foam from Hexcel. The carbon fiber composites were five-harness satin fabric and EPON 862/EPI CURE W laminates of 60v% fiber volume. The buckypapers were impregnated with EPON 862/EPI CURE W resin matrix and cured on the surface of the machined foam or carbon fiber composites. The buckypaper/foam and buckypaper/carbon fiber composite samples are shown in Figures 6 and 7, respectively. A total of eight composite samples with buckypaper (two for each type) were produced in the research.

Table 1. Electrical resistivity anisotropy ratios of the magnetically aligned buckypaper

		Res	lstivity	Test fo	r L-A-2 A	ligace Bu	ckypaper					
									Tester: Date:	Tr. 8/9/	1cy 2004	
No.	Sample No.	Direction	leagth. (cm)	Width (cm)	Thickness (cm)	Iffective Thickness (cm)	kesistivii - y (Q-cm)	Average	Mean	Std Dev.	C. Y.	Antsotrop
1	L-A-2-C-1	J (top)	2.945	0.649	0.0013	0.00098346	0.00157	1.558-03				
2	L-A-2-C-2	# (top)	2.978	0.653	0.0013	0.00104394	0.00158 0.00163	1.618-05	1.631-03	8.7864E-05	5.39%	
3	L-A-2-C-3	# (top)	2.954	0.614	0.0013	0.00101563	0.00172 0.00175	1.742-03				
4	L-A-2-C-4	⊥(tep)	2.225	0.686	0.0013	0.00096057	·0.00555 0.00563	5. 59E-05				3.062+00
5	L-A-2-C-5	⊥(top)	2.611	0.702	0.0013	0.00094348	8.00456 0.00465	4.612-03	4.998-03	0.00047564	9.54%	
6	L-4-2-C-6	L(top)	2.431	0.703	0.0013	0.0009679	0.00473	4:76E-03				
7	L-A-2-C-7	I (cat)	2.165	0.616	0.0013	0. 00 0 87386	0.00165 · 0.00159	1.62E-03				
8	L-A-2-C-8	I (cat)	2.165	0.607	0.0013	0.00091542	0.00188° 0.00180	1.841-03	1.698-03	0.00012323	7.31%	
,	L-A-2-C-9	f (cut)	2.199	0.685	0.0013	Q. 00097335	0.00160 0.00160	1.601-03				
10	L-A-2-C-10	L(cat)	3.084	0.552	0.0013	0.00103111	0.00592 0.00586	5. 891-03				3.496+00
11	L-A-2-C-11	L(cat)	3. 037	0.668	0.0013	0.00096361	0.00611	6.081-03	5.881-03	0.00018378	3.13X	
12	L-A-2-C-12	L(cat)	3.009	0.693	0.0013	0.00102763	0.00566 0.00568	5. 67E-03				

Table 2. Test samples of buckypaper/foam or carbon fiber composites

Sample #	Description	# BP Layers
1	Random BP / Foam - 1	2
2	Random BP / Foam - 2	2
3	Aligned BP / Foam - 1	2
4	Aligned BP / Foam - 2	2
5	Random BP / Carbon - 1	2
6	Random BP / Carbon - 2	2
7	Aligned BP / Carbon - 1	2
8	Aligned BP / Carbon - 2	2
9	Foam Control - 1	0
10	Foam Control - 2	. 0
11	Carbon Control - 1	0
12	Carbon Control - 2	0

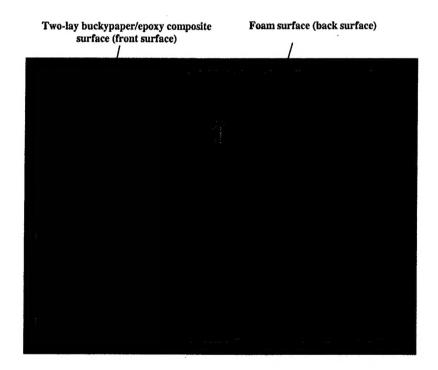


Figure 6. Buckypaper/ ROHACELL PMI foam sample (Front and back surface)

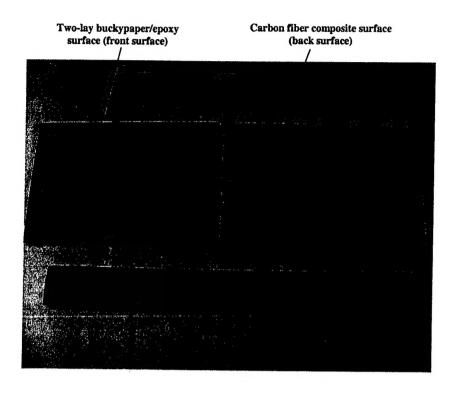


Figure 7. Buckypaper/carbon fiber composite sample (Front and back surface)

3.2 Tests and Analysis of EMI and Lightning Strike Protection Properties

Lockheed Martin Missiles and Fire Control – Orlando conducted the EMI and lightning striking tests of the samples.

3.2.1 EMI Tests and Results

Four different buckypaper composite samples and two control samples were tested to determine the EMI attenuation effectiveness of random and aligned buckypaper materials. All specimens measured 4" x 6". For the aligned buckypaper specimens, the carbon nanotubes were aligned along the 6" direction.

The testing method was conducted in accordance with MIL-STD-285 guidelines. An aluminum box with one open side was used. The dimensions of the open side panel were approximately that of the buckypaper composite test panels, plus a metallic grounding structure required to prevent radiation from entering the aluminum box through gaps or holes between the test panel and the box structure. The RF attenuation at specific frequencies between 200 MHz and 500 MHz from the external transmitted RF field and the received energy penetrating the test panel and sensed within the shielded box produced the actual measurement of EMI resistance of the test samples. The setup of EMI test is shown in Figure 8.

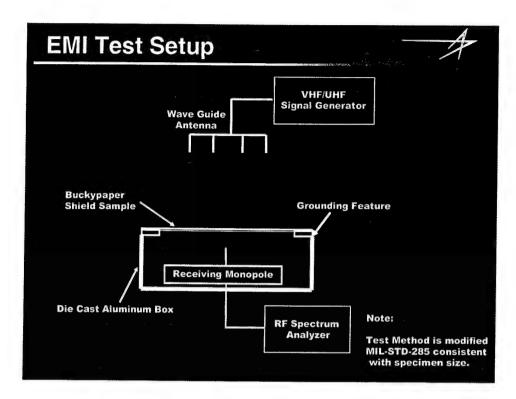


Figure 8. Setup of EMI tests

The test results of the buckypaper/foam composites and the foam control panel are shown in Figure 9. The graph shows the baseline radiation emitted/received as the top solid black line. The second line down on the graph (orange dashed line) represents the aluminum box with the foam control panel over the opening. The next line down (dotted blue) represents the result of the aligned buckypaper/foam composites. The difference between the foam control panel and the aligned buckypaper/foam composites represents the attenuation or EMI shielding effectiveness attributable to the aligned buckypaper material. This ranged from a minimum of 2 dB at 290 MHz to a max of 16 dB at 500 MHz. The average was 11 db of attenuation over the test range. The next line down on the graph (dashed-dotted fuscia) represents the results of the random buckypaper/foam. The difference between the foam control and the random buckypaper/foam composites represents the attenuation of the random buckypaper material. This ranges from a minimum of 12 db at 290 MHz to a maximum of 26 dB at several frequencies in the 455 to 500 MHz range. The average was 21 dB of EMI shielding over the test range. Considering the very thin thickness (two layers of 15~25 µm buckypapers) and less than 700mg SWNTs in the samples, the EMI shielding effectiveness was significant. The random samples had much better EMI shielding performance since the SWNTs are easier to form continuous networks in the random buckypapers than in the aligned buckypapers. In the magnetically aligned buckypapers, the SWNTs tried to become parallel to each other rather than forming continuous tube networks.

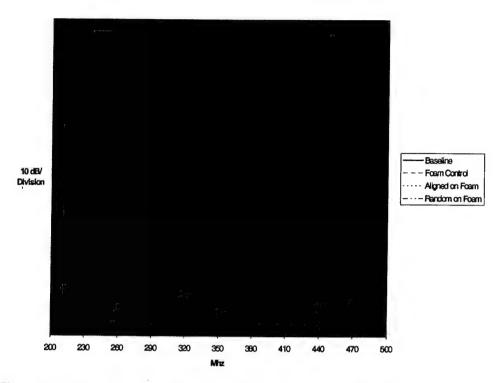


Figure 9. EMI test results of the SWNT buckypaper/foam composites

For the buckypaper/carbon fiber composite samples, the team did not observe any improvements in the EMI shielding performance, as shown in Figure 10. This graph shows the baseline radiation emitted/received as the top solid black line. The second line down on the graph (orange dashed line) again represents the aluminum box with the foam control panel over the opening. The difference between the baseline and the foam control panel represents the attenuation of the test setup.

The dashed green line on the graph represents the carbon/epoxy control specimen. The difference between the foam control and the control panel of the carbon fiber composites represents the attenuation attributable to the carbon/epoxy control. This ranged from a minimum of 26 dB at 215 MHz to a max of 40 dB at 500 MHz. Rough average was 35 dB of EMI shielding over the test range due to the samples having high carbon fiber loading (60v%). The dashed-dotted fuscia line on the graph represents the carbon/epoxy specimen with random oriented buckypaper. The difference between the foam control panel and this line represents the attenuation attributable to the carbon/epoxy with random oriented buckypaper. This ranged from a minimum of 22 dB at 215 and 290 dB MHz to a max of 41 dB at 500 MHz. The rough average was 32 db of shielding over the test range, which was a slightly lower that that of the control panel of carbon fiber composites. The dotted blue line on the graph represents the carbon/epoxy specimen with aligned buckypapers. The difference between the foam control and this line represents the attenuation attributable to the carbon/epoxy with aligned buckypaper. This ranged from a minimum of 23 dB at 290 MHz to a max of 38 dB at 500 MHz. The rough average was 32 dB of shielding over the test range, which was the same as the result of the random

buckypaper/carbon fiber samples. The EMI shielding performance of both the random and aligned buckypaper/carbon fiber samples were slightly lower than that of the carbon fiber control panel. The possible reason was due to the carbon fiber samples of high fiber loading (60v%) already having a high EMI attenuation and the conductivity of the buckypaper surface layer was lower than that of the carbon fiber control panels (see Section 3.2.2).

3.2.2 Test and Results of Lighting Strike Resistance

Four different buckypaper/carbon fiber and foam composite samples along with their control samples were tested to determine the impact of lightning strikes on random and aligned SWNT buckypapers. All specimens measured 4" x 6". For the aligned buckypaper specimens, the carbon nanotubes were magnetically aligned along the 6" direction.

The lightning current component, which is typically defined as a 6.4 x 70 microsecond double exponential current pulse for analysis purposes, applied current across the composite samples. A maximum current level of 20 kA can be achieved with the 6.4 x 70 microsecond waveshape, depending on the characteristics of the test article. The experiment setup developed at LTI, Lockheed Martin is shown in Figure 11.

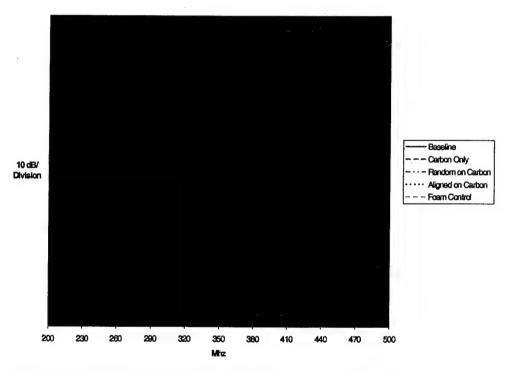


Figure 10. EMI test results of the SWNT buckypaper/foam composites

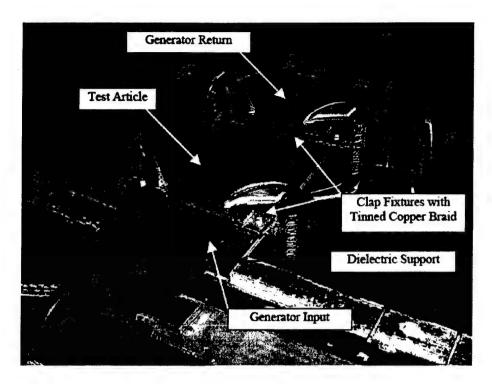


Figure 11. Experiment setup of lightning striking tests

Tests were conducted in the following manner:

- a) A 6 x 4" panel was installed in a conductive test fixture that provided full edge contact along two opposite sides of the panel. The two halves of the conductive test fixture were joined with insulated threaded rods such that conduction of the lightning currents was only across the panel under test. Threaded rods were used to clamp the test fixture to the panel.
- b) The end-to-end resistance of the panel under test was measured.
- c) The two halves of the test fixture were connected between the output terminals of a pulse generator such that the current would be conducted across the panel under test (along the long 6" direction). The pulse generator was capable of producing a nominal 6.4 x 70 microsecond current waveform.
- d) An initial current of 5 kA was applied to the panel and post-test panel resistance was measured. The panel was examined for signs of physical damage.
- e) If physical damage was noted, a second sample of the same panel configuration was installed and steps (b) through (d) were repeated at the same test current level. If no physical damage was noted, the current was increased an additional 5 kA and steps (d) and (e) were repeated until the maximum generator current limit of 20 kA was reached.

f) Steps (a) through (e) on each panel configuration were repeated for all the test samples. Figure 12 shows the lightning strike test in the research.

The test results are documented in Table 3. In the table, the "Generator Charge" column indicates voltage of the generator (in kV) applied to the sample and the "Ipk" column is the peak current (in amps) applied to the panel. The "Resistance" column shows the resistance (in ohms) before and after the current was applied to the panel.

The failure current for the samples was determined by visual evidence of arcing/flashing on the surface of the samples since no noticeable increase in resistance for any of the samples was observed. The buckypaper/foam samples were not subjected to the exact current outlined in the test procedure above since they could not handle the starting point of 5kA. The buckypaper/carbon fiber control specimen is the last group listed on the chart and shows a maximum current of 28kA with no evidence of failure.

The aligned and random buckypapers/foam samples showed relatively poor current carrying ability relative to the buckypaper/carbon fiber control sample before visual evidence of arcing was detected. The resultant current carrying abilities are on the order of 200 amps for aligned buckypaper/foam and 700 amps for random buckypaper/foam samples. The random oriented buckypaper samples carried slightly more current than the aligned samples but probably not enough to be significant. The aligned and random buckypapers/carbon fiber panels showed better performance than that of the foam samples but still short of the carbon fiber control. The random buckypaper/carbon fiber sample showed contact sparking at approximately 24kV.

One possible reason that no improvement in the lighting strike resistance of the buckypaper samples was observed could be due to the relatively lower surface conductivity, which can be seen in Table 3. This is because the buckypaper/EPON 862 surface layers had a higher resin content (>50w%) than the carbon fiber laminates. A resin rich area may exist on the surface of the buckypaper samples since buckypaper materials have an extremely low permeability due to fabrication [1~5]. The team believes that a further increase of buckypaper composite conductivity could improve lighting strike resistance properties.

Table 3. Results of lightning strike tests

No. Charts 2004 Applied Current LII 3045 Test No. Charge Hortz Nett Ratio Ipk Action Integral Sample No. Charge Hortz Vert RAV A X10 ⁴ A ² s Test 1 2.75 10 0.2 2 100 0.000059 Metal Bar 1 2.75 40 0.2 2 176 Aligned Foam #1 1 11.1 100 0.2 2 176 Aligned Foam #1 2 1.5 10 0.2 2 176 Aligned Foam #1 1 1.1 100 0.2 2 480 Aligned Foam #1 2 2.75 100 0.2 2 480 Random Foam #1 3 5.5 100 0.2 2 480 Random Foam #2 1 1.1 1.0 0.2 2 2.0 Random Foam #1 5 1.0 0.2					تَدُ	ghtning Test of (Lightning Test of Carbon Nanotube Bucky Paper	Bucky Paper				
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85 100 0.2 2 28,400 0.047	8	71	5	0.5	2	24,000	0.018	Graphite Control		0.0452		
	8	88	<u>8</u>	0.2	~	28,400	0.047	Graphite Control		0.0429		

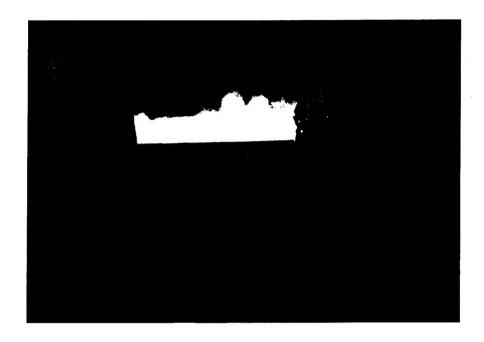


Figure 12. Lighting strike testing

4. Conclusions

In this exploratory research project, a unique technical approach was developed to incorporate SWNTs buckypaper materials into conventional fiber-reinforced and foam composite structures for improved EMI and lightning strike protection properties. The EMI shielding and lightning strike attenuation properties of the carbon fiber-reinforced composites and foam structures with the surface layer of SWNT Buckypaper nanocomposite were preliminarily characterized. Four types of the designed EMI/lightning strike testing composite samples with SWNT buckypapers were successfully produced. Each sample had two layers of random or magnetically aligned buckypapers 15~25 µm thick. Each sample size was 6"x4"x1/8" with approximately 700mg purified SWNTs covering its surface. The results show that the foam structures with the buckypaper surface can achieve an average 21 dB of EMI shielding over the test range of 455 to 500 Mhz, compared to the control panel of pure foam structure. The results also show that the random buckypaper samples exhibited better EMI shielding properties. However, there was a slight reduction of EMI shielding of the carbon fiber composites with the buckypaper surface, compared to the controlled panel. For the lightning strike resistance, no visible improvement was observed. Further improvements in electrical conductivity of the SWNT buckypaper composites are vital for utilizing SWNTs to achieve EMI and lightning strike resistance properties for composite structures.

5. Acknowledgements

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Dr. James Brooks Mr. Olivier Marietta-Tondin Ms. Liao (Ashley) Yu-Hsuan

6. References

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